AD-A039 120 CONSTRUCTION ENGINEERING RESEARCH LAB (ARMY) CHAMPAI--ETC F/G 11/2
REVISED OPERATIONS GUIDE FOR A CHEMICAL TECHNIQUE TO DETERMINE --ETC(U)
APR 77 P A HOWDYSHELL

UNCLASSIFIED CERL-TR-M-212 NL

1 OF 1
ADA039120

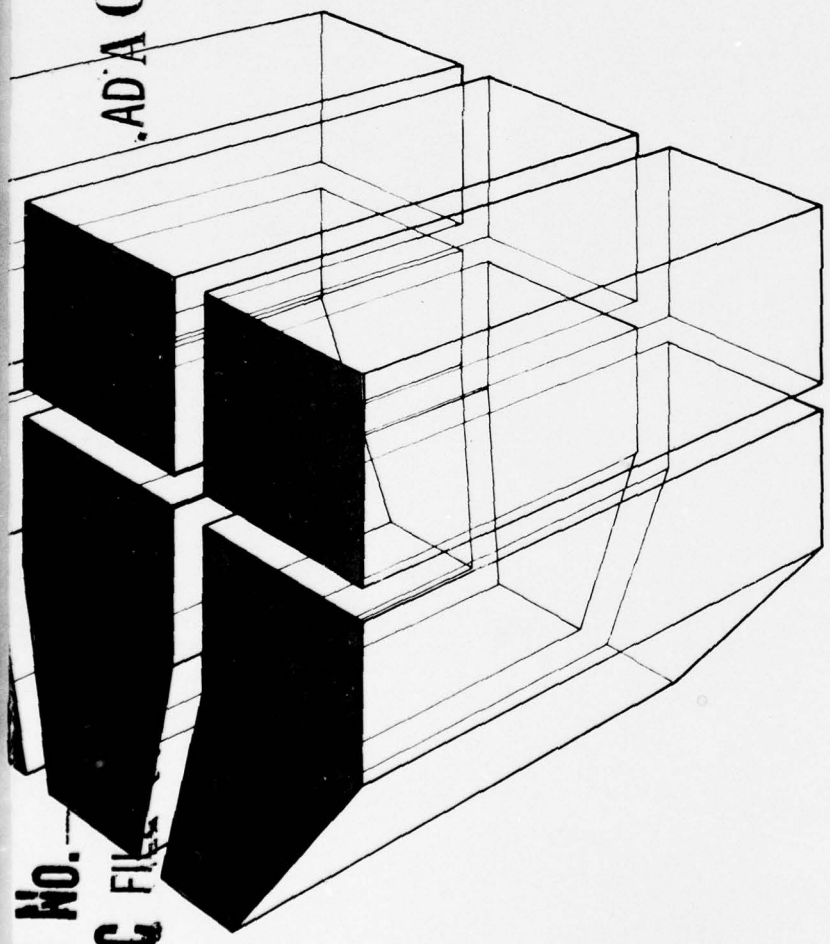
END

DATE
FILMED
5-77

construction
engineering
research
laboratory

TECHNICAL REPORT M-212
April 1977

REVISED OPERATIONS GUIDE FOR A CHEMICAL TECHNIQUE TO DETERMINE WATER AND CEMENT CONTENT OF FRESH CONCRETE



by
P. A. Howdyshell

CERL

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

REPORT DOCUMENTATION PAGE	READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER: CERL-TR-M-212	2. GOVT ACCESSION NO. / 3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle): REVISED OPERATIONS GUIDE FOR A CHEMICAL TECHNIQUE TO DETERMINE WATER AND CEMENT CONTENT OF FRESH CONCRETE	5. TYPE OF REPORT & PERIOD COVERED: FINAL rept.
	6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s): P. A. Howdyshell	8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS: CONSTRUCTION ENGINEERING RESEARCH LABORATORY, P.O. Box 4005, Champaign, IL 61820	10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS: 4A763734DT08-04-002
11. CONTROLLING OFFICE NAME AND ADDRESS	12. REPORT DATE: April 1977
	13. NUMBER OF PAGES: 35 (38 p.)
14. MONITORING AGENCY NAME & ADDRESS *(if different from Controlling Office)*	15. SECURITY CLASS. *(of this report)*: Unclassified
	15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT *(of this Report)*

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT *(of the abstract entered in Block 20, if different from Report)*

18. SUPPLEMENTARY NOTES

Copies are obtainable from National Technical Information Service
Springfield, VA 22151

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

fresh concrete
concrete strength
water content

405279 DB

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

This operations guide provides information needed to set up and operate the U. S. Army Construction Engineering Research Laboratory/Kelly Vail (CERL/K-V) system for determining water and cement content of fresh concrete. The guide describes the system's capabilities (including its capability to estimate potential concrete strength), limitations, and accuracy, and details the required equipment, reagents, and procedures. A guide for analyzing test results and a description of a mobile miniaturized field unit are presented.

DD FORM 1 JAN 73 1473 EDITION OF 1 NOV 65 IS OBSOLETE

UNCLASSIFIED

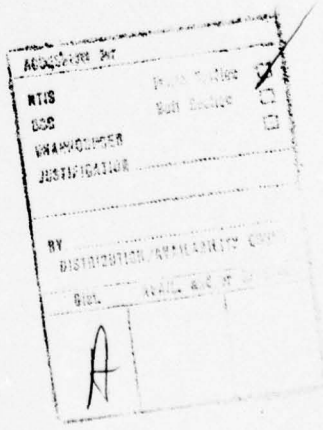
SECURITY CLASSIFICATION OF THIS PAGE *(When Data Entered)*

FOREWORD

This work was performed for the Directorate of Military Construction, Office of the Chief of Engineers (OCE), under Project 4A763734DT08, "Military Construction and Engineering Field Development"; Task 04, "Construction Systems Development"; Work Unit 002, "Rapid Testing-Plastic PCC." The applicable QCR is 1.06.003. Mr. R. Liebhardt is the OCE Technical Monitor.

The research was conducted by the Construction Materials Branch (MSC), Materials and Science Division (MS), U. S. Army Construction Engineering Research Laboratory (CERL).

Mr. P. A. Howdyshell is Chief of MSC, and Dr. G. R. Williamson is Chief of MS. COL J. E. Hays is Commander and Director of CERL and Dr. L. R. Shaffer is Technical Director.



CONTENTS

TABLES

FIGURES

REVISED OPERATIONS GUIDE FOR A CHEMICAL TECHNIQUE TO DETERMINE WATER AND CEMENT CONTENT OF FRESH CONCRETE

1 INTRODUCTION

Background

This guide provides the information needed to set up and operate the U.S. Army Construction Engineering Research Laboratory/Kelly-Vail (CERL/K-V) system for determining water and cement content of fresh concrete. The system relies on chloride ion titration for determining water content and calcium titration for cement content. Tests have proven the system to be rapid (less than 15 minutes), simple, robust, and field-worthy.

The CERL/K-V system is an outgrowth of a system originally proposed by Kelly and Vail of the Greater London Council.[1] The original system (K-V) relied on chloride ion titration to determine water content, and flame photometry (calcium signature) to determine cement content. Extensive tests have proven the K-V system to be rapid (15 minutes) and accurate enough to estimate the strength potential of fresh concrete.[2] However, the constant subdued light requirement of the flame photometer necessitates packaging the system for field use in either a pick-up truck camper or a small trailer.[3]

Tests conducted by CERL in early 1976 indicated that the calcium signature required in the K-V cement test could be determined by simple titration using an EDTA (ethylenediaminetetra-acetate) solution in the presence of a buffer and an eriochrome black T indicator. Results indicate that the EDTA titration system is as accurate and rapid as the flame photometer and much simpler and less costly. In addition, substitution of the calcium-EDTA titration for the flame photometer reduces the field packaging requirements.

Purpose

This guide provides a detailed description of equipment, equipment packaging, reagents, and procedures for operating the CERL/K-V system. The system's capabilities, limitations, and accuracy, including its capability to estimate potential concrete strength, are discussed.

Mode of Technology Transfer

The information in this report is applicable to the Corps of Engineers' *Handbook for Cement and Concrete,* and has potential application as an American Society for Testing and Materials (ASTM) standard test method.

2 CERL/K-V TEST SYSTEM EQUIPMENT AND OPERATIONS

Equipment

Tables 1 and 2 list the required equipment and related cost for the CERL/K-V water content and cement content tests, respectively. Figures 1 and 2 illustrate the equipment.

Several of the items can be replaced by other equipment that performs the same function. The polyethylene fixed volume dispensers (item 14, Table 1) can be replaced by automatic dispensing glass pipettes (Figure 3). In the packaged field test setup, the universal mixer (item 5, Table 1) is replaced by a laboratory-fabricated end-over-end mixer driven by a 1/70-hp (10.7 W) gear motor.

The approximate 1976 cost for the items listed in Table 1 (including extras for breakage) is $1241. If the universal mixer (item 5, Table 1) is replaced by the laboratory-fabricated end-over-end mixer and gear motor, the cost of items listed in Table 1 is $831. Excluding items 1, 2, 3, and 17 in Table 2, which are duplicates of items in Table 1, the approximate 1976 cost of the items listed in Table 2 (including extras for breakage) is $799. Thus, the total equipment cost for the CERL/K-V test system is approximately $2040 with the universal mixer or $1630 with the end-over-end mixer. These costs do not include the costs of cleanup equipment or storage bottles for extra reagents, but neither is a significant expense.

[1] R. T. Kelly and J. W. Vail, "Rapid Analysis of Concrete," *Concrete,* (April 1968), pp 140-145.

[2] P. A. Howdyshell, *Laboratory Evaluation of a Chemical Technique to Determine Water and Cement Content of Fresh Concrete,* Technical Report M-97/AD784055 (U.S. Army Construction Engineering Research Laboratory [CERL], July 1974); P. A. Howdyshell, "Correlating Kelly-Vail Test Results to the Strength Potential of Fresh Concrete," *Rapid Testing of Fresh Concrete,* Conference Proceedings M-128/ADA009702 (CERL, May 1975); and P. A. Howdyshell, *Operations Guide–Water and Cement Content of Fresh Concrete,* Technical Report M-177/ADA022697 (CERL, February 1976).

[3] Howdyshell, *Operations Guide.*

Equipment Packaging and Other Requirements for Field Use

For the CERL/K-V system to be field-worthy, it must be packaged in a small, portable, self-contained unit that can be set up for use rapidly. Figure 4 depicts one layout where all equipment except the washing machine and the sieves is housed in a 48 x 30 x 14 in. (1.2 x 0.8 x 0.4 m) box. The unit is self-contained, with enough reagents for eight to ten tests (a normal day's run), and requires only 110 to 115 V of AC current and tap water to be operational. If not locally available, the electric current can be supplied by a 1.5 kW alternator, and the tap water by an appropriately sized storage tank (Figure 5).

The size of the packaged system allows it to be easily transported in the back of a pick-up truck, and set up to operate from the truck's tailgate (Figures 6 and 7). The top of the box is hinged so that it is in a vertical position during testing. Most of the glassware is either permanently attached to the top of the box, or to a rod which stores horizontally and is simply placed vertically for use (Figure 8). All tube connections that must be made for setting up and tearing down disconnect quickly, so the time required for setting up the unit for use, or tearing down for transporting, is normally less than 15 minutes.

Figures 9, 10, and 11 are detailed drawings of the packaged system. These drawings and the photographs should enable a user to build the system. The total cost of assembling the units (including carpenter and machinist time) should be about $1600. Including the $1630 equipment cost, an entire assembled unit should cost $3230.

Water Content Test

Reagents

The reagents required for conducting the water content test are:

1. Sodium chloride (NaCl) solution, approximately 0.5 normal (*N*) in tap water

2. Silver nitrate ($AgNO_3$) solution, approximately 0.5*N* in distilled water

3. Potassium thiocyanate (KCNS) solution, approximately 0.05*N* in distilled water

4. Nitrobenzene

5. Ferric alum solution (saturated)

6. 50 percent nitric acid solution in tap water.

The reagents required to produce solutions 1, 2, and 3 can be purchased in crystalline or powder form, or as premade solutions of the proper strength. Appendix A describes the method for making all six solutions. Table 3 lists the quantities and costs of solutions and reagents required for 100 tests.

The concentration of solutions 1, 2, and 3 must be in the ratio of 1:1:10 (third significant digit) and should be checked by titration. If it is determined that solutions 1, 2, and 3 are not exactly in the 1:1:10 ratio, and if facilities are not available to rebalance them, the procedure described in Appendix B for recomputing the volume-normality relationships of the solutions and the related water content of the concrete sample should be used.

Procedure

The method of determining water content is based on the intermixing of available water in fresh concrete with an aqueous solution. The technique consists simply of adding 500 mℓ of a 0.5*N* sodium chloride solution to a 1-kg concrete sample, intermixing the two, and determining the chloride concentration of the intermixed solution using the Volhard back-titration method. If the concrete contains chlorides from other sources, the procedure requires use of both a sample and a blank (500 mℓ of distilled water added to a 1-kg concrete sample).

The steps required to conduct a CERL/K-V water content test are described below and outlined in Appendix C. The outline in Appendix C should be posted near the equipment so operators can refer to it as needed.

1. Obtain a 6- to 8-kg sample of fresh concrete, mix sample to insure homogeneity, and weigh out two 1-kg subsamples to the nearest gram. Place one 1-kg sample in a wide-mouth jar; using a volumetric flask, add 500 mℓ of distilled water. Secure the lid on the jar. This sample is the blank required for estimating chlorides in the concrete itself.

2. Place the second 1-kg sample in another wide-mouth jar, add 500 mℓ of the approximately 0.5*N* sodium chloride solution, and secure the lid.

3. Secure the two jars in the end-over-end mixer, and mix for 3 minutes.

4. Remove the jars from the mixer and allow contents to settle for 3 to 5 minutes.

5. Using a volumetric pipette, withdraw 50 mℓ of clear sample solution and place it in a 500-mℓ conical beaker. Using the automatic pipette, add 50 mℓ of approximately 0.5*N* silver nitrate solution to the clear sample solution. Using the fixed volume dispensers, add 10 mℓ of 50 percent nitric acid solution (two shots from a 5-mℓ dispenser); 5 mℓ of ferric alum (one shot from a 5-mℓ dispenser); and 2 mℓ of nitro-benzene (one shot from a 2-mℓ dispenser). Shake well (by hand) for a few seconds.

6. Determine chloride strength by initially adding 50 mℓ (automatic pipette) of 0.05*N* potassium thiocyanate solution to the solution in the conical beaker. To complete the titration, swirl the contents of the beaker while adding the 2-mℓ shots of 0.05*N* potassium thiocyanate solution from a fixed volume dispenser. When the first permanent reddish-brown color appears, the end point has been reached. Stop titration and note the number of potassium thiocyanate shots added.*

7. Using a volumetric pipette, transfer 50 mℓ of the blank solution to a conical beaker. Add 10 mℓ of silver nitrate solution (automatic pipette), 10 mℓ of 50 percent nitric acid solution (fixed volume dispenser), 2 mℓ of nitrobenzene (fixed volume dispenser), and 5 mℓ of ferric alum solution (fixed volume dispenser). Shake well. Titrate (100-mℓ burette) using the 0.05*N* potassium thiocyanate solution, until the first permanent reddish-brown color appears.

The blank is calculated as follows:

$$y' = 100 - x \qquad \text{[Eq 1]}$$

where y' = millilitres of the thiocyanate equivalent to chloride in the blank
x = quantity of thiocyanate solution required to reach end point (mℓ) in step 7.

The volume of thiocyanate equivalent (y') obtained from the blank solution is added to the volume of thiocyanate used in the sample solution. The percent water is calculated as follows:

$$\% \text{ Water} = \frac{50z}{500 - z} \qquad \text{[Eq 2]}$$

where z = total volume of thiocyanate (y' plus 50 mℓ plus two times the number of thiocyanate shots required in step 6).

The curve generated by Eq 2 (Figure 12) should be used directly in the field. However, Eq 2 applies only when the silver nitrate, sodium chloride, and potassium thiocyanate reagents are in the ratio of 1:1:10. As indicated in the section on reagents, Appendix B contains equations for determining water content when the silver nitrate, sodium chloride, and potassium thiocyanate solutions are not in the exact 1:1:10 ratio. It is recommended that the equations be used only when it is impractical to adjust the solution strengths.

If the concrete being tested does not contain chlorides, use of the blank should be discontinued after the initial determination.

Appendix D describes a modified water test procedure which is identical to the standard procedure except that it uses one-half the volume of silver nitrate to determine the chloride strength of the intermixed sample solution. This is significant, since the price of silver nitrate is both unstable and relatively high compared to the other reagents (Table 3).

Cement Content Test

Reagents

The reagents required for conducting the cement content test are:

1. A 5 percent nitric acid solution made by adding 5 mℓ nitric acid (specific gravity 1.42) to 95 mℓ of tap water.

2. Tap water.

3. Standard cement solution equivalent to a 1-kg sample of concrete containing 24 percent cement by weight (240 g) prepared by running a standard cement content test (outlined in the cement test procedure) on 240 g of cement. The cement used in preparing the cement standard must be the same as that used in the concrete being tested. It is recommended that it be obtained from the stockpile being used to produce the concrete.

4. A 0.01 molar *(M)* solution of disodium EDTA containing 3.72 g of EDTA per litre. Distilled water

*The final titration (step 6) can be accomplished using a 100-mℓ burette, which will give a more exact endpoint. The choice of dispenser or burette is the option of the operator.

should be employed in making up the solution. Since the EDTA solution is slightly reactive with the calcium and magnesium present in ordinary (soda) glass, it should be stored in polythene bottles if possible.

5. Eriochrome black T indicator prepared by dissolving 0.5 g of the dyestuff in 25 mℓ of triethanolamine. The solution has a limited stability (several months), with the quality of the end point being influenced by the age of the indicator solution. It is recommended that new indicator solutions be prepared at least every 2 months.

6. Ammonia-ammonium chloride buffer solution (pH = 10) made by adding 142 mℓ of concentrated ammonia solution (specific gravity 0.88 to 0.90) to 17.5 g of reagent-grade ammonium chloride, and diluting to 250 mℓ with distilled water. Since the ammonia in the buffer solution evaporates rapidly, the solution should always be covered when not in use.

The cement standard and tap water are used in calibrating the cement test. A cement content titration (outlined in the cement test procedure, steps 10 through 12) is run on the cement standard solution and the tap water. The millilitres of EDTA required for the tap water represent zero percent cement and the millilitres of EDTA required for the cement standard represent 24 percent cement in the concrete. All unknown cement contents are linearly proportional. A calibration curve is then plotted for the cement standard; Figure 13 is a typical calibration curve for the cement test.

Procedure

The CERL/K-V method of determining cement content is based on three assumptions: (1) when agitated, cement can be dispersed in water and held uniformly in suspension so that a representative sample can be obtained; (2) stirring without external heat will produce a quantitative solution of cement in nitric acid; and (3) the calcium content of the cement solution can be determined by titration, with a readily perceptible end point.

The steps required to conduct a cement content test are described below and outlined in Appendix E. The outline in Appendix E should be posted near the test equipment so operators can refer to it as needed.

1. Fill the washing machine to the manufacturer's fill mark on the tank with 37.6 ℓ of tap water (Figure 14). Place the nested sieves on the washing machine.

2. Charge the automatic pipettes with their appropriate reagents—300 mℓ of tap water and 100 mℓ of 5 percent nitric acid solution.

3. Remix the 6- to 8-kg concrete sample (step 1, water test procedure) to insure homogeneity, and weigh out 1 kg of fresh concrete to the nearest gram.

4. Transfer the weighed concrete to the sieves over the washing machine. Turn on the washing machine's recirculating pump and agitator and wash the residue from the 1-kg sample container into the washing machine using the jet of water from the recirculating pump hose.

5. Wash the plus no. 4 aggregate carefully, using the jet of water from the recirculating pump hose. After cleaning (about 1½ minutes), remove the no. 4 sieve.

6. Wash the plus no. 50 aggregate for about 1½ minutes.* Squeeze the end of the large bore hose to force the cement suspension to flow through the small bore tubing.

7. Rapidly release the large bore hose, allowing the cement suspension to flow through it, and connect the end of the small bore tubing to the 125-mℓ linked pipette. Squeeze the large bore hose again to direct the cement suspension into the pipette. When the pipette is full, switch off the lower pipette tap and release the large hose.

8. Run the 125-mℓ aliquot of the suspension into the mixer cup. Wash out the automatic pipette using the 100 mℓ of 5 percent nitric acid solution contained in the automatic pipette fitted above the sample pipette. The material washed out will also run into the mixer cup. Add 300 mℓ of tap water from the third automatic pipette.

9. Fix cup to stirrer and stir for 3 minutes to insure complete solution of cement.

10. After stirring is completed, pipette off 25 mℓ of the resulting cement solution and place in a conical beaker.

*If calcareous fines are present, it is recommended that an additional no. 100 wash sieve be nested below the no. 50. When both the no. 50 and no. 100 sieves are used, the no. 50 sieve should be removed after its 1½-minute wash cycle, and the aggregates retained on the no. 100 sieve should be washed for about 1½ minutes.

11. Add 10 mℓ of buffer solution (ammonia-ammonium chloride) and 4 to 6 drops of indicator solution (eriochrome black T) to the conical beaker. The buffer must be added before the indicator solution. Shake well (by hand) for a few seconds.

12. Titrate (100-mℓ burette) using the 0.01-*N* EDTA solution. The end point is reached when the solution turns from a wine color to a pronounced blue.* Record the millilitres of EDTA required to reach the end point. Using the previously calibrated graph, relate millilitres of EDTA to percent cement in the sample.

3 LIMITATIONS AND CAPABILITIES

Limitations

The standardized 1-kg sample is the only significant operational limitation of either the water or cement content tests. This sample size limits applicability of the methods to concretes containing maximum aggregates of 1½ in. (38.1 mm) or less. This restriction, which is required to insure that the sample is representative of the concrete being tested, can be removed by modifying the procedures and using larger concrete samples. Applying the restriction that the largest aggregate must weigh less than 10 percent of the sample weight, 2, 2½, and 3 in. (50.8, 63.5, 76.2 mm) aggregates would require samples of approximately 2, 4, and 6 kg respectively.

In the water test procedure, larger concrete samples would require only that the volume of the sodium chloride solution used for intermixing be increased proportionately with sample size. For example, a 4-kg concrete sample would require 2 ℓ of sodium chloride solution. The only restriction on increasing the sample size of the cement test is that the wash sieves do not become overloaded with aggregates, clog, and prevent the circulating water from re-entering the washing machine.

The cement test has a second limitation related to the fact that the technique does not measure cement per se, but all calcium in the concrete that passes the finest sieve nested above the washing machine (either a no. 50 or no. 100 sieve). Thus, aggregates passing the sieve nest and containing calcium are also detected by the calcium EDTA titration as cement. Tests on both coarse and fine calcareous (limestone) aggregates indicate that only fine aggregates cause significant interference; interference caused by coarse aggregates is normally less than 5 percent. The interference caused by the fine calcareous aggregates depends on the finest sieve used and varies from 20 to 40 percent for the no. 50 sieve, to 5 to 15 percent for the no. 100 sieve.

Capabilities

Accuracy of the water determination method is limited by (1) the volume of sodium chloride titrated, (2) the related type and size of the potassium thiocyanate dispenser used in determining the titration end point, and (3) the perceptibility of the end point reaction. For the standard 50-mℓ sodium chloride titration sample, a 2-mℓ constant volume potassium thiocyanate dispenser will have an error of 0.25 percent water (approximately 1 gal of water per cu yd of concrete [4.9 ℓ/m³]). The modified 25-mℓ sodium chloride titration sample with the 1-mℓ constant volume potassium thiocyanate dispenser has the same accuracy.

If greater accuracy is desired, a conventional 100-mℓ burette is used to dispense the potassium thiocyanate. Its limiting accuracy is the operator's ability to perceive the titration end point. For the reagent strengths used, the end point reaction can be detected with a sensitivity of about 0.5 mℓ. Thus, the perception sensitivity in the end point reaction produces an error in measuring water content of about 0.06 percent water for the 50-mℓ sodium chloride titration sample and 0.12 percent water for the 25-mℓ sodium chloride titration sample.

The accuracy of the cement determination is limited by (1) the volume of cement solution titrated (25 mℓ) and (2) the perceptibility of the end point reaction. Repetitive tests on the same sample have indicated that the error in perceiving the end point is normally 0.5 mℓ; thus, for the 25-mℓ cement solutions, which require 40 mℓ of EDTA solution to reach the end point, the limiting error in estimating cement content is about 0.3 percent cement (15 lb cement per cubic yard of concrete [8.9 kg/m³]).

The actual operational accuracies of the CERL/K-V water and cement content tests have been determined in extensive laboratory and field tests. These tests have indicated that the average error in the CERL/K-V determined water contents is from 4 to 6 percent, and cement contents, 6 to 8 percent. The laboratory tests also indicated that the CERL/K-V determined water contents are more closely related to the free water than

*It is recommended that operators not wear tinted (sun) glasses during the calcium titration process. Tinted glasses can alter perception of the wine to blue end point.

the total water present in the concrete. One field test has indicated that the ferric alum end point in the water content test is difficult to perceive at temperatures below 55°F (11.6°C) and becomes nearly impossible at temperatures below 40°F (4.4°C). But these results have not been confirmed by laboratory testing. The tests have also indicated that both the water and cement content test procedures are, for all practical purposes, insensitive to such parameters as aggregate moisture conditions, mix proportions, cement type*, length of mix time, and aggregate type (water only).

The water and cement content tests can be run concurrently by a single operator, requiring only 10 to 15 minutes to complete. The operational cost per test is $2.10 for reagents and $13.00 for labor (Table 3).

Experimental data from the CERL/K-V evaluation indicate that the water and cement contents obtained can be used to predict the potential strength of fresh concrete with a reliability equal to that of predictions based on actual mix proportions. For the normal range of aggregate types and sizes and a given cement type, air entrainment appears to be the other common material parameter significantly influencing strength.** Thus, the CERL/K-V system used in conjunction with an air content test provides a rapid means (less than 15 minutes) for determining the strength potential of a given concrete.

The accuracy (80 percent confidence) of the strength predictions normally varies between 600 and 700 psi (4.1 and 4.8 MPa); field and laboratory accuracies are equal.

4 ANALYSIS OF TEST RESULTS

Comparison to Mix Design Values

The water and cement contents determined by the CERL/K-V method should be compared to the mix design values. If the CERL/K-V and mix design values vary by less than 10 percent, it can be assumed that the CERL/K-V system is operating properly. If the results vary by more than 10 percent, a second complete CERL/K-V test should be run. If the two tests agree closely and indicate a variation of greater than 10 percent, one of three things has occurred: (1) the concrete sample is not representative of the bulk (indicating poor mixer efficiency and/or segregation); (2) the mix being batched is not the same as the mix design; or (3) the CERL/K-V system has malfunctioned.

Several checks can be run to determine whether either part of the CERL/K-V system is malfunctioning. For the water content test, the reagents can be checked by adding a known volume (80 to 100 mℓ) of water* to the wide-mouth jar and running a standard water test on the pure water sample. If the results are within 5 percent of the mix design, the reagents' strengths are in the proper 1:1:10 ratio. If a water test blank to determine whether chlorides are present in the concrete was not run when the water content of the samples was determined, it should be done. If the tests indicate that the reagents' strengths are in the proper ratio and that chlorides are not present in the concrete or have been blanked out, and the CERL/K-V determined water contents deviate from the mix designs, then the concrete tested was not representative of the bulk, or the mix delivered was not the same as the mix design.

The following steps are recommended to determine whether variation between CERL/K-V cement results and mix design values is caused by equipment malfunctions:

1. A standard cement content test to determine cement equivalency of the aggregate should be run on aggregate blanks of both the fine and coarse aggregate. Each aggregate blank should weigh (in grams) 10 times the weight percent of the aggregate in the mix. The cement content of the concrete sample minus the cement equivalency of the aggregate should be within 10 percent of the cement content of the mix.

2. If the error in cement content after step 1 modifications is still greater than 10 percent, a new cement standard should be prepared, and the quantity of EDTA required to reach end point for a 25-mℓ sample of the standard should be checked. The quantity of EDTA required for a 25-mℓ sample of the tap water should also be checked. The tap water sample should be the same as that used to fill the washing machine and the 300-mℓ automatic pipette. If the upper and lower

*This may not be true for the blended hydraulic cements, ASTM C-595. The calcium contents of types IP, P, IS, and S are allowed to vary widely.

**In some cases, water-reducing agents or plasticizers influence concrete strength beyond their direct effect on water/cement ratios.

*Distilled water should be used if available. If not, a blank should also be run.

titration end points have shifted more than ½ mℓ, a new calibration curve should be prepared.

If these steps indicate that the procedure is operating properly and the CERL/K-V determined cement contents deviate from the mix design, either the concrete tested was not representative of the bulk, or the mix delivered was not the same as the mix design.

Determination of Concrete Strength

Figure 15 shows a typical relationship between the CERL/K-V water/cement ratios, air content, and 28-day compressive strength. This relationship was developed from tests on Type I and Type II cements with air-entraining agents being the only admixture present. The accuracy (80 percent confidence band) for estimating strength normally varied between 600 and 700 psi (4.1 and 4.8 MPa). Thus, the strength of a concrete sample will be less than 700 psi (4.8 MPa) below the value predicted from Figure 15 only 10 percent of the time.

If other cement types that have different strength-gain characteristics are used, or other additives or admixtures that influence the strength-gain characteristics of the concrete are present, the relationship in Figure 15 will be in error. When the strength to water/cement ratio and air content relationship is known from trial mix data for a given mix, this data can be used to modify the relationship in Figure 15. If it is found that the Figure 15 strength predictions average a certain percentage of the actual strengths, this percentage relationship can be used to estimate unknown strengths. Where time and data allow, this percentage-type modification can be used to improve the accuracy of the strength predictions for all mixes. The resulting accuracy (80 percent confidence band) based on this type of individual mix calibration will normally decrease the error in strength estimates to 500 to 600 psi (3.4 to 4.1 MPa).

5 ADDITIONAL INFORMATION ON SYSTEM

Since this guide may not answer all questions from potential users, CERL will provide technical assistance to any Corps of Engineers facility wanting to set up and use the CERL/K-V test system. This includes assistance in procuring equipment, training operators, analyzing results, and troubleshooting. For any type of assistance on the CERL/K-V system, contact:

Department of the Army
Construction Engineering Research Laboratory
ATTN: P. A. Howdyshell
P. O. Box 4005
Champaign, IL 61820
Telephone: 958-7224 (FTS) or 217-352-6511,
ext. 224 (commercial)

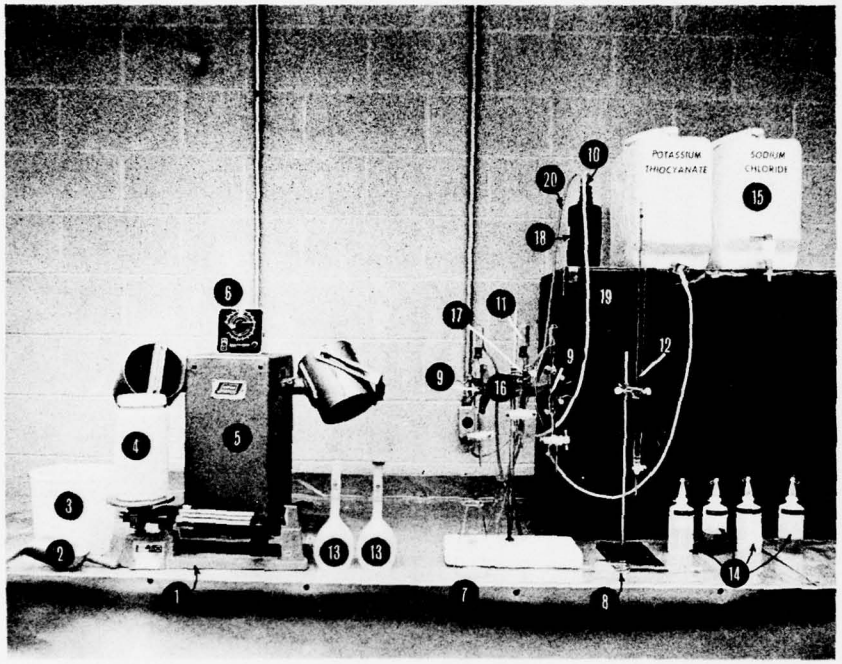


Figure 1. CERL/K-V water test equipment.

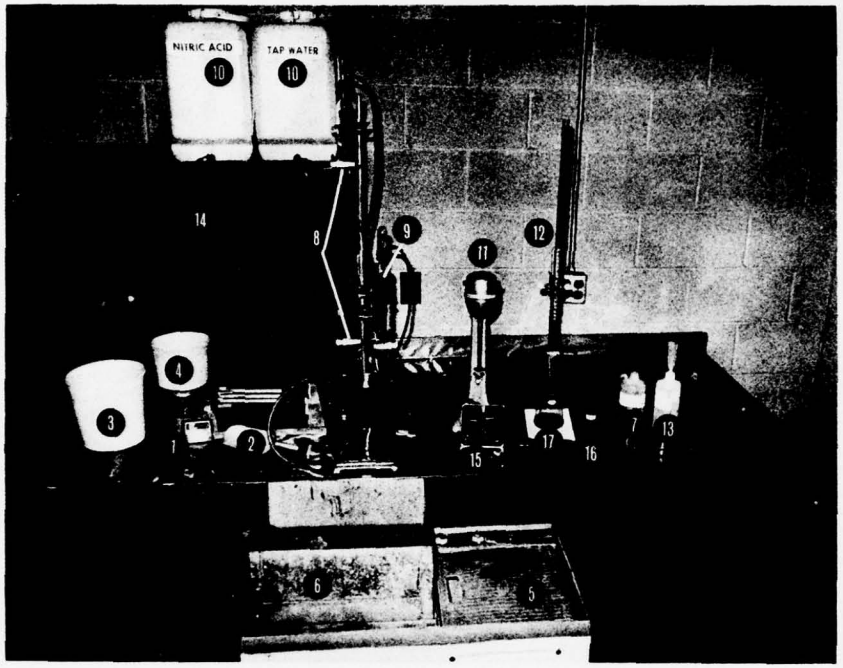


Figure 2. CERL/K-V cement test equipment.

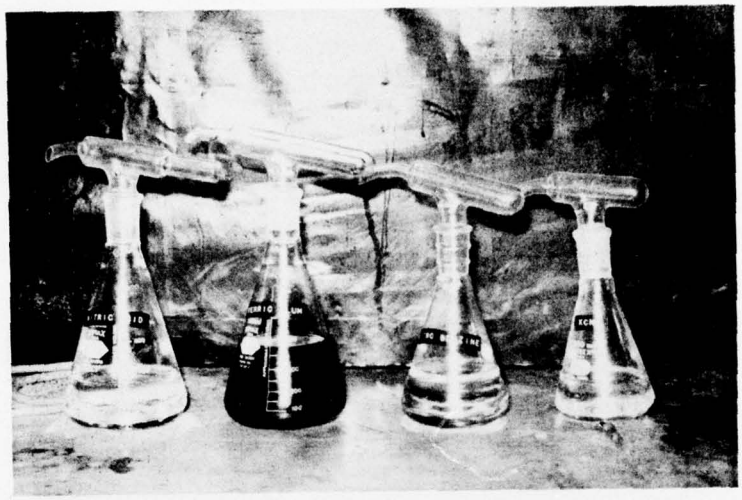


Figure 3. Automatic dispensing pipettes.

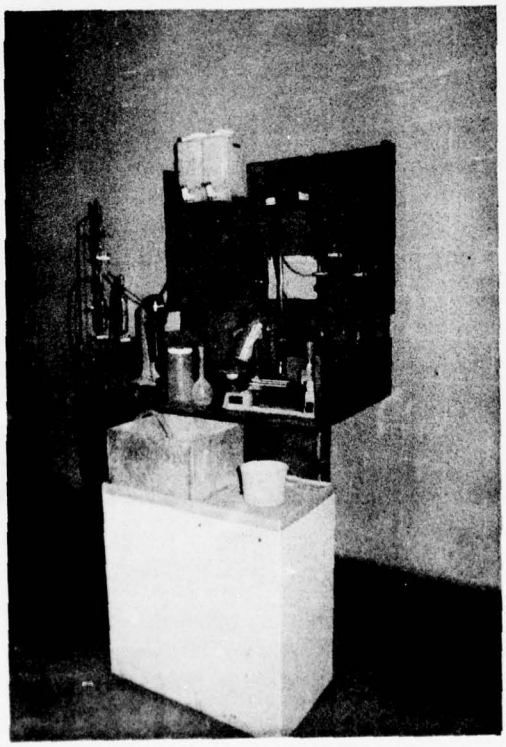


Figure 4. Packaged CERL/K-V unit.

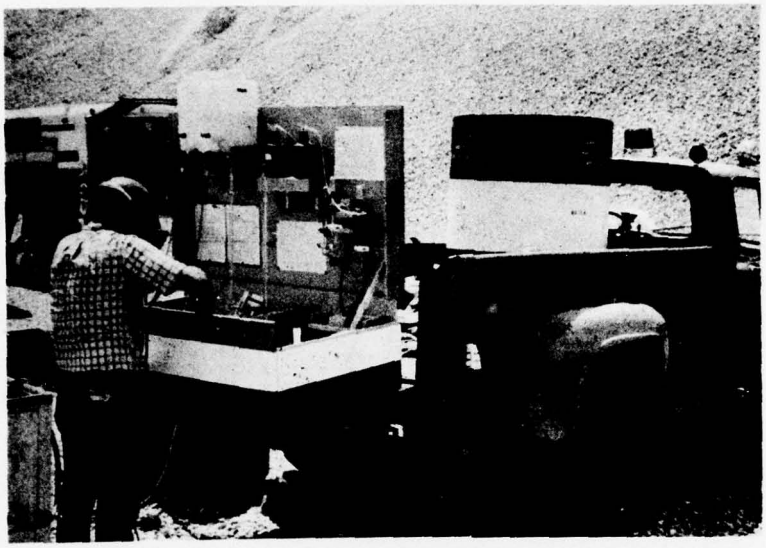


Figure 5. Typical water storage tank for use with CERL/K-V system.

Figure 6. Typical field transport of packaged unit.

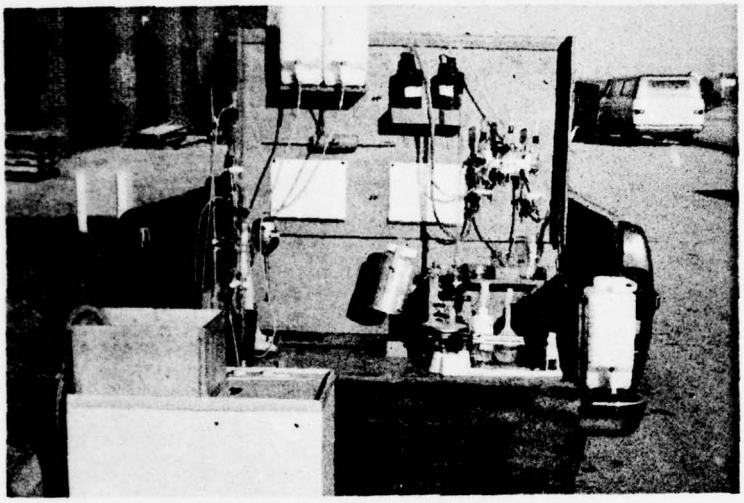


Figure 7. Packaged unit ready for field use.

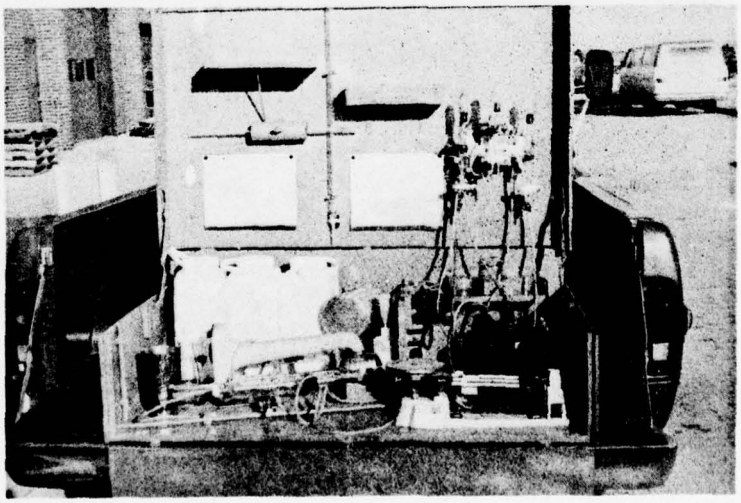
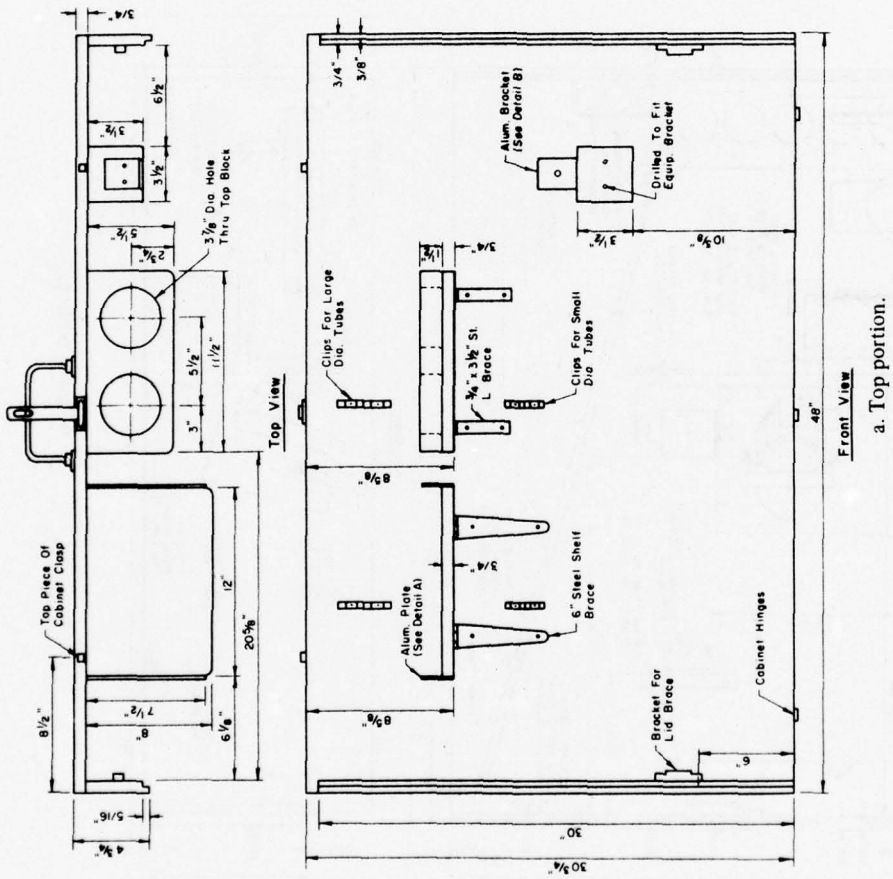
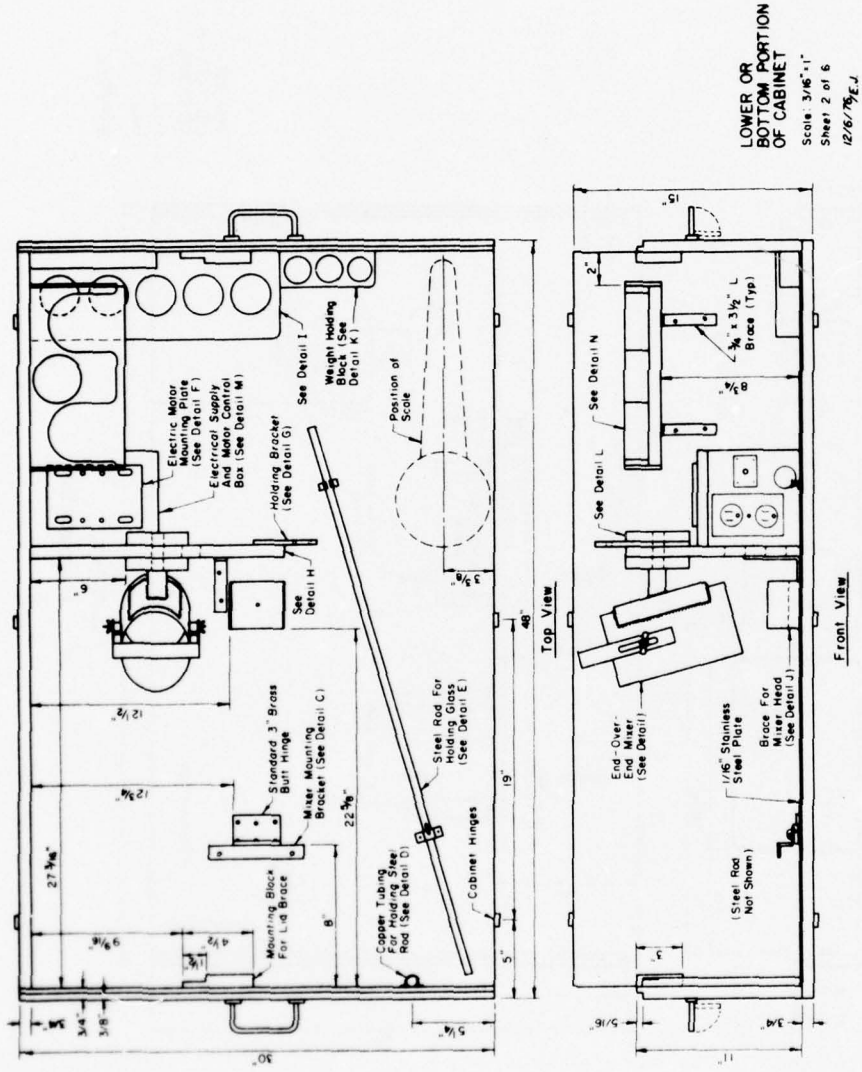


Figure 8. Packaged unit ready for transportation. Note location of reagent bottles and glassware.



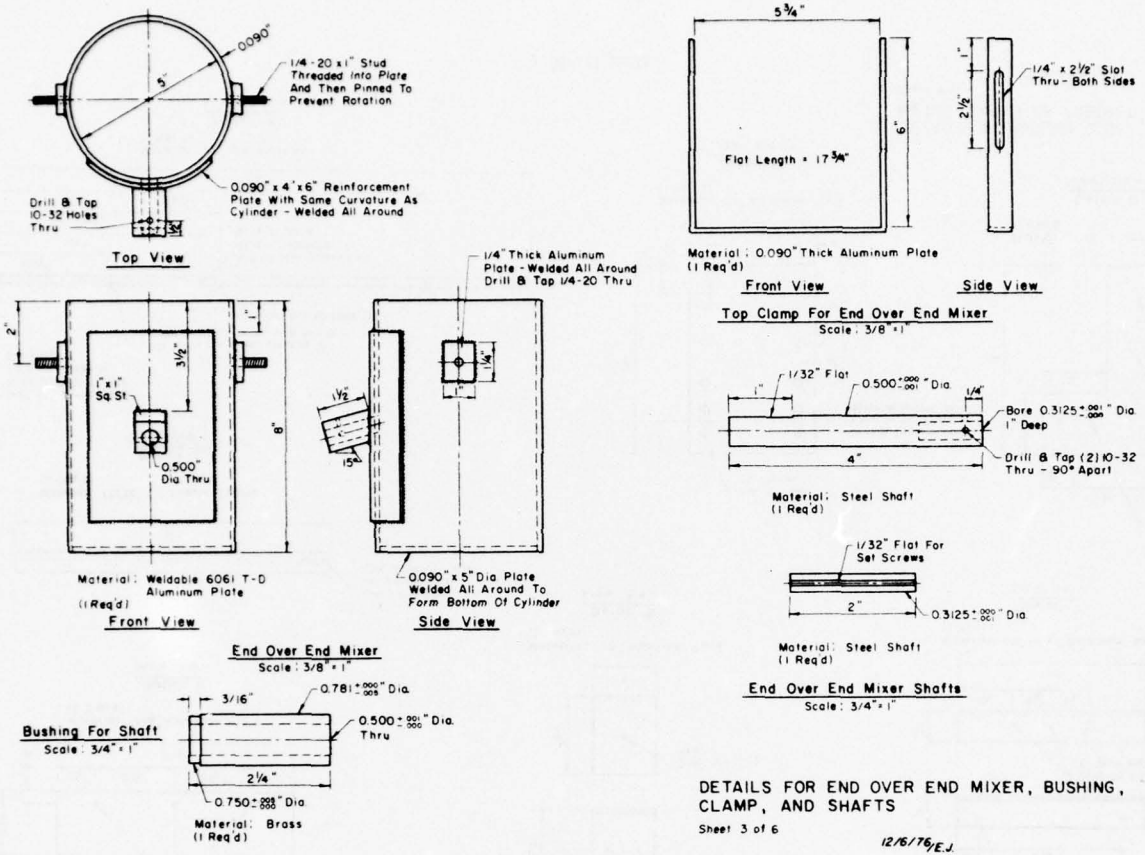
a. Top portion.

Figure 9. Top and lower portions of CERL/K-V packaged system. SI conversion factor: 1 in. = 25.4 mm.



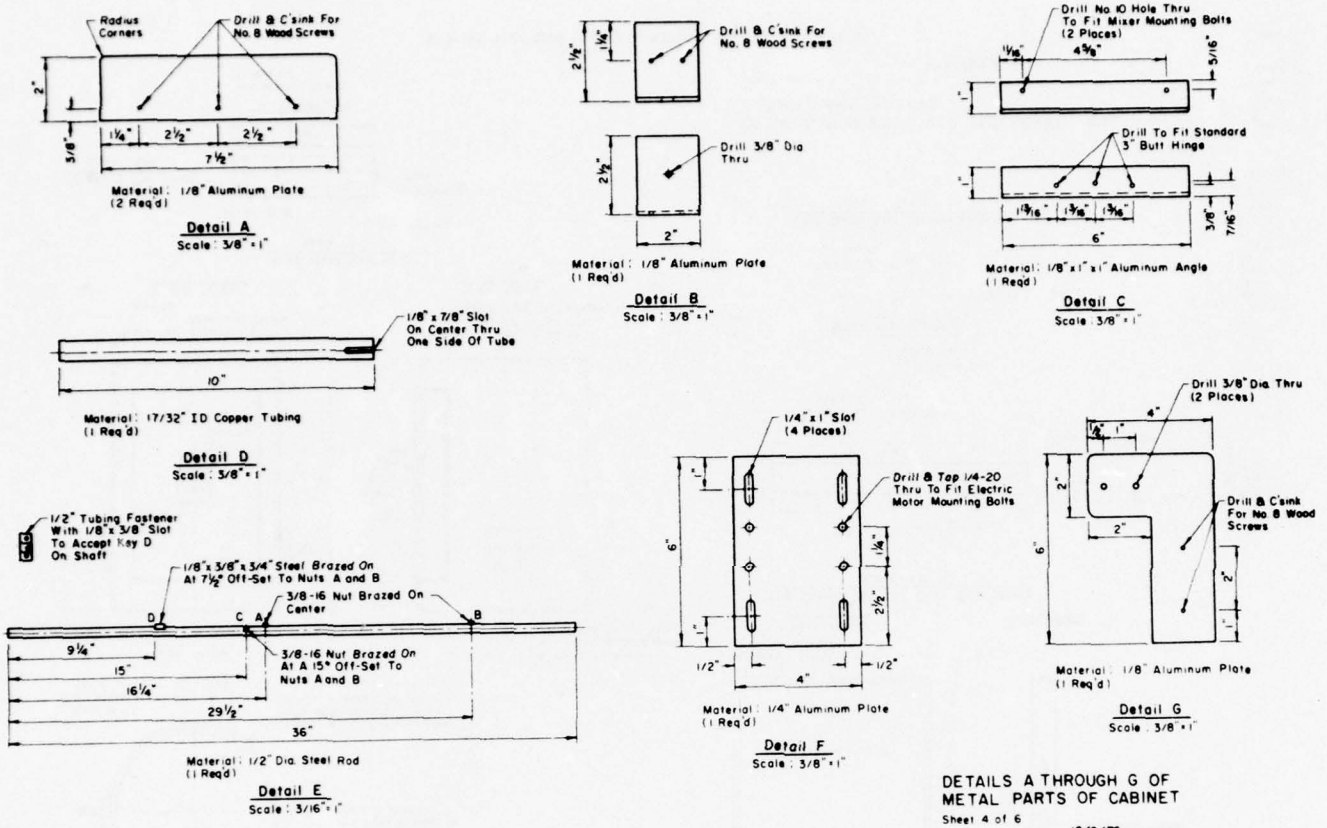
b. Lower portion.

Figure 9 (cont.)



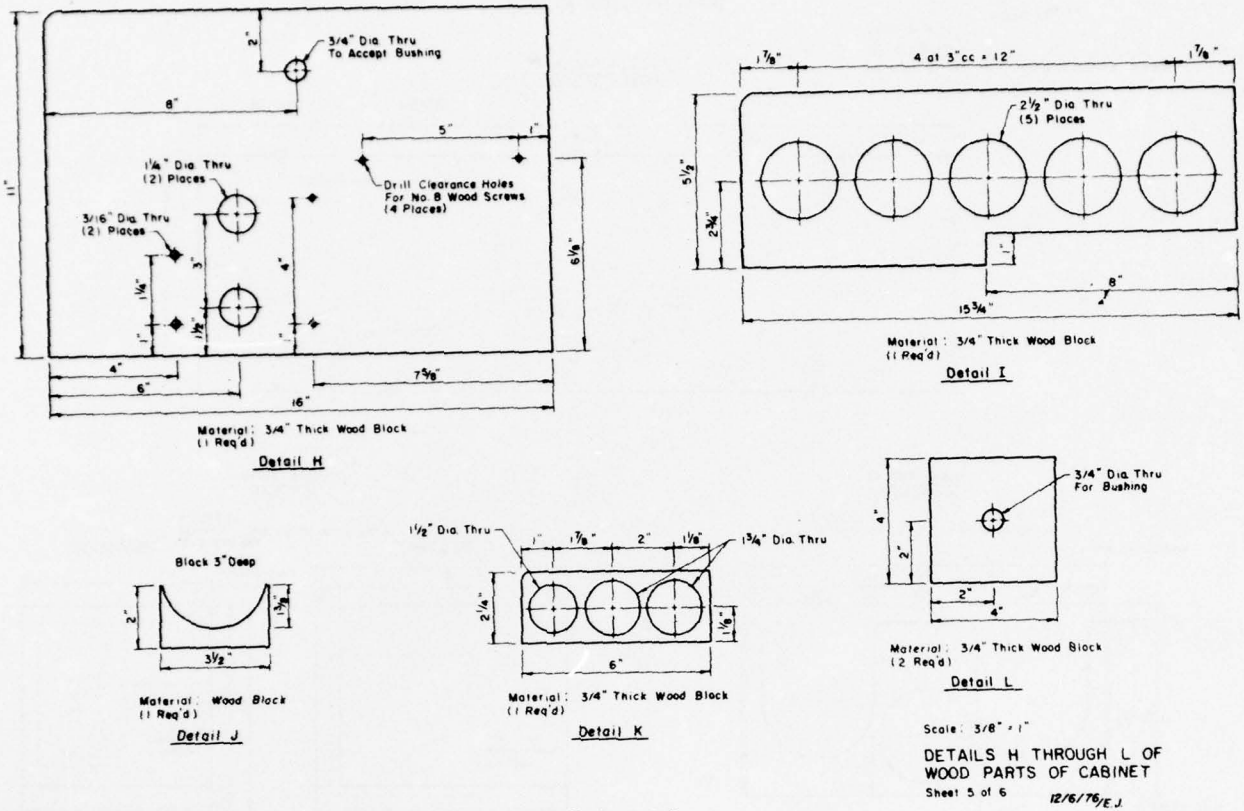
a. End-over-end mixer, bushing, clamp, and shafts.

Figure 10. Details of A through G. SI conversion factor: 1 in. = 25.4 mm.



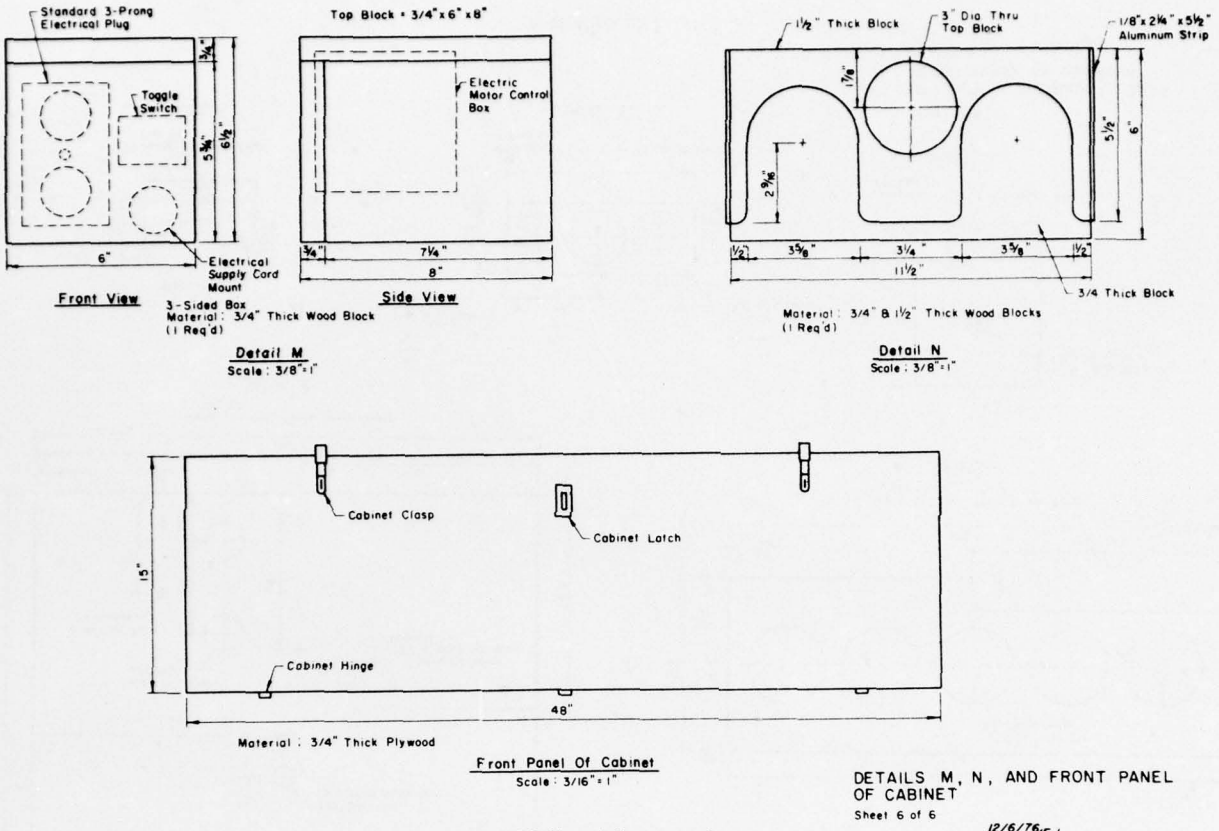
b. Metal parts.

Figure 10 (cont.)



a. H. through L.

Figure 11. Details of H through N and front panel. SI conversion factor: 1 in. = 25.4 mm.



b. M, N, and front panel.

Figure 11 (cont.)

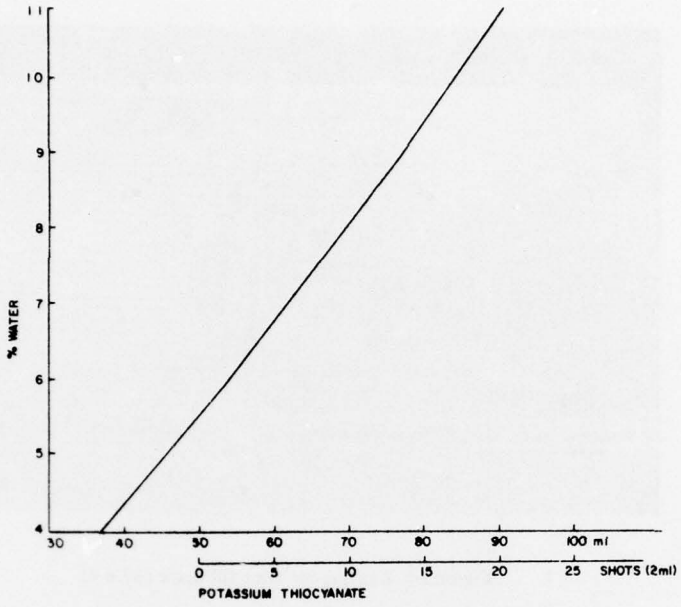


Figure 12. Water content vs. potassium thiocyanate.

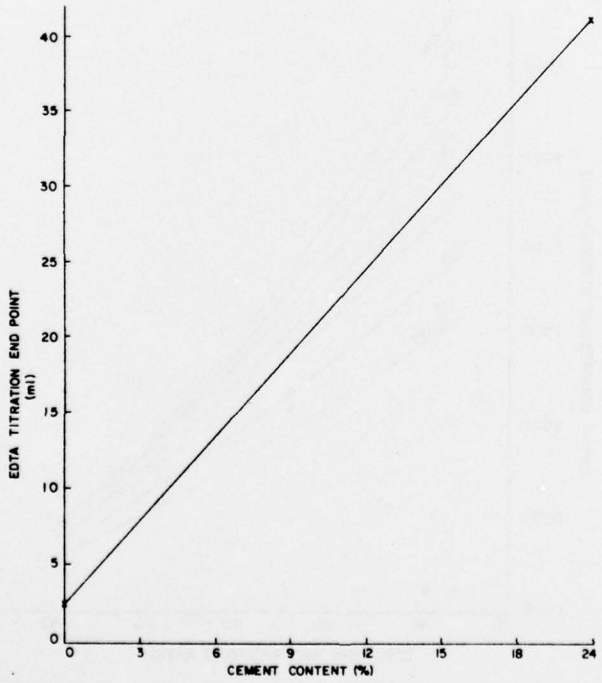


Figure 13. Typical cement content vs. EDTA titration end point.

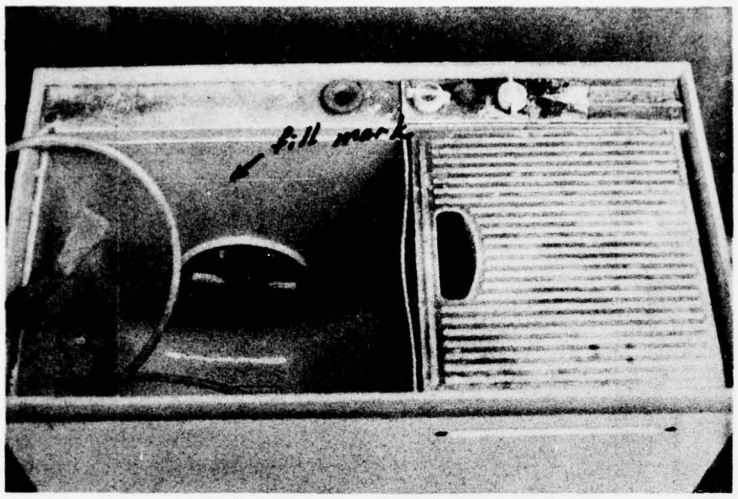


Figure 14. Washing machine fill mark (37.6 ℓ).

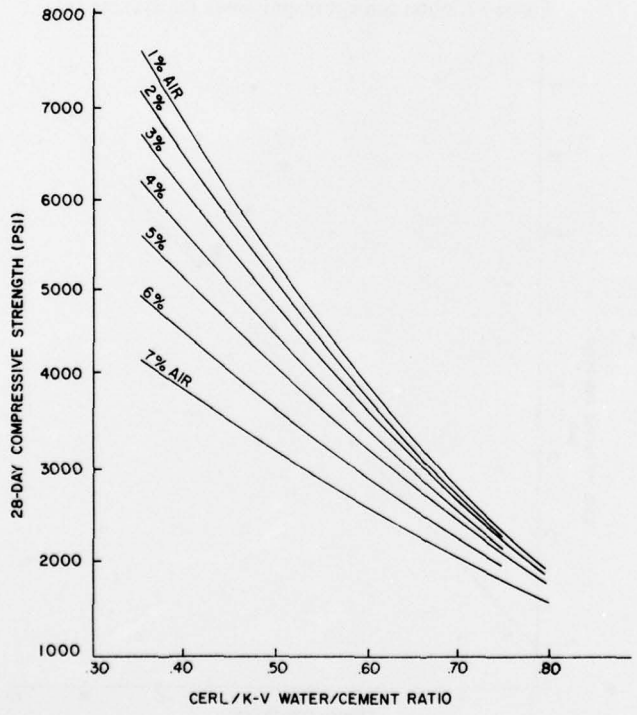


Figure 15. Strength vs. water/cement ratio and air. SI conversion factor: 1 psi = 6.9 kPa.

Table 1
Water Test Equipment

Item	Title	Quantity*	Description	Sources	Cost (approx.) Per Unit	Cost (approx.) Total**
1	Scales	1	Triple-beam (+2000 g capacity), 0.1-g sensitivity	Equipment suppliers for concrete and soils testing labs	$ 60	$ 60
2	Hand scoop	1	One-piece, cast-aluminum; bowl size approximately 3 1/4 × 5 × 2 in. deep (82.6 × 127. × 50.8 mm)	Same as item #1	$ 4	$ 4
3	Sample tub	1	5-qt (4.7 ℓ) polyethylene tub	Domestic food freezer supplier goods	$ >1	$ >1
4	Wide-mouth jar	2	1/2-gal (1.9 ℓ), polyethylene wide-mouth jar with screw closure and lid	Laboratory glass supplier	$ 3	$ 6
5	Universal mixer†	1	Double-ended shaft, driven at 50 rpm by 1/4-hp (0.19 kW) motor (AC)	Laboratory equipment supplier	$ 860	$ 860
6	Timer	1	0 to 15 min. industrial timer switch with automatic reset (115 V, 60 cycle)	Laboratory equipment supplier	$ 45	$ 45
7	Conical beaker 500 mℓ	2	Narrow-mouth conical beaker, 500-mℓ capacity. To allow for breakage, a shelf-pack of 12 is normally ordered	Laboratory glass supplier	$ 10	$ 10 (shelf-pack)
8	Pipette, 50 mℓ	1	Volumetric pipette, glass, class A, 50-mℓ capacity. To allow for breakage, a shelf-pack of 12 is normally ordered	Laboratory glass supplier	$ 20	$ 20 (shelf-pack)
9	Automatic pipette, 50 mℓ	2	Automatic pipette with teflon plug, glass, 50-mℓ capacity. To allow for breakage, two cases with two each are normally ordered	Laboratory glass supplier	$ 36	$ 72 (case containing two)
10	Rubber stoppers No. 6	2	Rubber stoppers, no. 6; one with two holes and one with three holes	Laboratory glass supplier	$ >1	$ >1
11	Automatic pipette, 10 ml	1	Automatic pipette with teflon plug, glass, 10-mℓ capacity. To allow for breakage, one case containing two is normally ordered	Laboratory glass supplier	$ 33	$ 33 (case of 2)
12	Burette, 100 mℓ	1	Acrylic body, teflon plug. Class B accuracy, 100-mℓ capacity	Laboratory glass supplier	$ 22	$ 22
13	Volumetric flasks, 500 mℓ	2	Polypropylene, volumetric flasks, 500-mℓ capacity	Laboratory glass supplier	$ 9	$ 18
14	Fixed volume dispenser (a) 2 mℓ (b) 5 mℓ	2 2	Fixed volume dispenser of polyethylene and polypropylene measuring chamber; two 2-mℓ and two 5-mℓ dispensers required	Laboratory glass supplier	$ 7.50 $ 7.50	$ 15 $ 15
15	Carboys	1	Rectangular aspirator carboys with spigot, 2-gal (7.6 ℓ) capacity, linear polyethylene	Laboratory glass supplier	$ 22	$ 22
16	Double burette clamp	1	Double burette holder clamp, nonferric alloy die casting, nickel-plated	Laboratory glass supplier	$ 6	$ 6
17	Utility clamp	1	Utility clamp, three-pronged grip, vinylized jaws	Laboratory glass supplier	$ 6	$ 6
18	Amber bottles	2	Narrow-mouth amber bottles. Boston rounds from amber polypropylene (32 oz or 0.91 kg). Normally purchased in packages of six bottles.	Laboratory glass supplier	$ 9	$ 9 (package of 6)
19	Rubber tubing 1/4 in. (6.4 mm)	10 ft (3.05 m)	Amber rubber latex tubing (standard wall 1/16 in. or 1.6 mm) sold in 96-ft (29.26 m) lengths	Laboratory glass supplier	$ 9	$ 9 (96-ft unit)
20	Glass tubing 7 mm OD	3	Glass tubing 7 mm OD × 10 in. (254 mm) long (standard wall)	Laboratory glass supplier	$ >1	$ >1

* Quantity required to conduct tests.

**Cost estimates based on quantities including breakage allowance.

† If the 1/70 hp (10.7 W) gear motor and locally fabricated end-over-end mixer are used, their cost is about $450.

Table 2
Cement Test Equipment

Item	Title	Quantity*	Description	Sources	Cost (approx.) Per Unit	Cost (approx.) Total**
1	Scales	1	Triple-beam scale (+2000 g capacity) 0.1 g sensitivity	Equipment suppliers for concrete and soils testing labs	$ 60	$ 60
2	Hand scoop	1	One-piece, cast-aluminum, bowl size approximately 3 1/4 × 5 × 2 in. (82.6 × 127. × 50.8 mm)	Same as item #1	$ 4	$ 4
3	Sample tub, 5 qt (4.7 ℓ)	1	5-qt (4.7 ℓ) polyethylene tub	Domestic food freezer supplier goods	$ 1	$ 1
4	Sample tub, 2 qt (1.9 ℓ)	1	2-qt (1.9 ℓ) polyethylene tub	Same as item #3	$ 1	$ 1
5	Washing machine	1	Domestic portable washing machine; must have a smooth interior, side-mounted impeller, and a recirculating pump and hose. Recirculating hose to be fitted with a t-piece for connecting to the automatic pipette. Working capacity of tub, 37.6 ℓ	Domestic appliance store	$ 110	$ 110
6	Sieve nest	1	Rectangular steel frame 15 5/16 × 12 5/16 × 8 in. (388.9 × 312.7 × 203.2 mm) deep. A no. 50 sieve at the bottom and a no. 4 sieve at midheight. The sieve should be removable†	Sieves purchased from equipment suppliers for concrete testing laboratories. Frame would be locally fabricated	$ 100 (sieve) $ 100 (frame)	$ 100 $ 100
7	Wash bottle	1	Wash bottle, polyethylene, 50 mℓ	Laboratory glass supplier	$ 2	$ 2
8	Linked pipettes	1 set	A 125 mℓ glass pipette (with automatic leveling devices), fitted with a three-way tap. To it is attached a 100-mℓ automatic pipette (three-way tap) that can empty its contents through the 125 mℓ pipette. To allow for breakage, two sets are normally ordered	Laboratory glass supplier (not a shelf item)	$ 80	$ 160
9	Automatic pipette	1	A 300-mℓ automatic pipette, three-way tap teflon stopcock, glass. To allow for breakage, two are normally ordered	Laboratory glass supplier	$ 45	$ 90
10	Carboys 2 gal (7.6 ℓ)	2	Rectangular aspirator carboys with 2 gal (7.6 ℓ) capacity linear polyethylene	Laboratory glass supplier	$ 22	$ 44
11	Stirrer	1	Stirrer (milk-shake type) with stainless steel containers	Laboratory glass supplier	$ 100	$ 100
12	Burette, 100 mℓ	1	Acrylic body teflon plug, class B accuracy, 100-mℓ capacity	Laboratory glass supplier	$ 22	$ 22
13	Fixed volume dispenser, 5 mℓ	1	Fixed volume dispenser of polyethylene and polyproplene measuring chamber	Laboratory glass supplier	$ 7.50	$ 7.50
14	Rubber tubing 1/2 in. (12.7 mm) ID	20 ft (6.1 m)	Amber rubber tubing (standard wall), sold in 96-ft (29.3 m) lengths	Laboratory glass supplier	$ 10 (96-ft unit)	$ 10
15	Timer	1	0 to 15 min industrial timer switch with automatic reset (115 V, 60 cycle)	Laboratory equipment supplier	$ 45	$ 45
16	Dropping bottle	1	Polyethylene, 30-mℓ narrow mouth bottle and dropping pipette	Laboratory glass supplier	$ 1	$ 1
17	Conical beaker, 500 mℓ	1	Narrow mouth conical beaker, glass, 500-mℓ capacity. To allow for breakage, a shelf-pack of 12 is normally ordered	Laboratory glass supplier	$ 10 (shelf-pack)	$ 10
18	Utility clamp	1	Utility clamp, three-pronged grip, vinylized jaws	Laboratory glass supplier	$ 6	$ 6
19	Pipette (25 mℓ)	1	Volumetric pipette, glass, class A, 25-mℓ capacity. To allow for breakage, a shelf-pack of 12 is normally ordered (not shown in Figure 2)	Laboratory glass supplier	$ 20 (shelf-pack)	$ 20
20	Suction bulb	1	Rubber suction bulb for the 25-mℓ pipette (not shown in Figure 2)	Laboratory glass supplier	$ 3	$ 3

* Quantity required to conduct test.

**Cost estimates based on quantities including breakage allowances.

† If calcareous sands are to be encountered, it is recommended that a no. 100 sieve be nested below the no. 50.

Table 3
Operational Cost for Reagents and Labor

Reagent Solutions	Solution Quantities				Reagent Quantities		Cost	
	1 Test		100 Tests		100 Tests		100 Tests	
Water Test	Standard	Modified	Standard	Modified	Standard	Modified	Standard	Modified
NaCl – 0.5*N*	500 mℓ	500 mℓ	50.0 ℓ	50.0 ℓ	1460 g	1460 g	$ 2.05	$ 2.05
$AgNO_3$ – 0.5*N*	60 mℓ	25 mℓ	6.0 ℓ	3.5 ℓ	509 g	509 g	$ 192.03	$ 112.05
KSCN – 0.05*N*	185 mℓ	142 mℓ	18.5 ℓ	14.2 ℓ	90 g	69 g	$ 2.86	$ 2.19
HNO_3 - 50%	20 mℓ	20 mℓ	2.0 ℓ	2.0 ℓ	1 ℓ (1.43 specific gravity)	1 ℓ (1.43 specific gravity)	$ 3.53	$ 3.53
Ferric Alum	10 mℓ	10 mℓ	1.0 ℓ	1.0 ℓ	500 g (ferric ammonium sulfate)	400 g (ferric ammonium sulfate)	$ 4.40	$ 4.40
Nitrobenzene	4 mℓ	4 mℓ	400 mℓ	400 mℓ	–	–	$ 2.61	$ 2.61
						Subtotal	$ 207.48	$ 126.83

Cement Test	Solution Quantities		Reagent Quantities	Cost
	1 Test	100 Tests	100 Tests	100 Tests
EDTA	40 mℓ	4 ℓ	15 g disodium EDTA	$ 0.60
Eriochrome Black T	>0.5 mℓ	>50 mℓ	0.8 g dyestuff 50 mℓ triethomolomine	$ 0.55
Ammonia-Ammonium Chloride Buffer	10 mℓ	1 ℓ	568 mℓ ammonia 70 g ammonium chloride	$ 1.90
			Subtotal	$ 3.05

Labor: 45 min/test at GS-9 rate ($15,977/annum) plus 100% overhead = $11.52/test = $1152./100 tests
Total CERL/K-V Operating Cost = $1302.53/100 standard
= $1281.88/100 modified tests

APPENDIX A:

PREPARATION OF SOLUTIONS FOR WATER CONTENT TESTS

1. Standard Sodium Chloride – 0.5*N*

Dry sodium chloride in oven at 110°C for a minimum of 4 hours. Then place sodium chloride sample in desiccator and cool to room temperature. Accurately weigh out 29.230 g of dry sodium chloride and place in 1000-mℓ volumetric flask. Add distilled water and dissolve salt. Fill to mark on flask with distilled water, and agitate to obtain uniform solution. This is the primary standard and is used to check the normality of the silver nitrate solution.

2. Silver Nitrate Solution – 0.5*N* (approx)

Weigh out 255.0 g of silver nitrate and dissolve in distilled water. Make up 3 ℓ. Standardize against the 0.5*N* sodium chloride solution using the Mohr titration technique explained later in this appendix.* Keep track of the millilitres of solution used so the total weight of silver nitrate can be computed after titration is complete. Compute normality of silver nitrate by the following equation:

$$N_{Ag}\, V_{Ag} = N_{Cl}\, V_{Cl} \qquad \text{[Eq A1]}$$

where N_{Ag} = unknown normality of silver nitrate solution

V_{Ag} = volume (mℓ) of silver nitrate solution required to reach titration end point

N_{Cl} = volume of sodium chloride solution used to reach titration end point

If the resulting normality of the silver nitrate solution is greater than 0.50, the solution must be diluted and the following equation applied:

$$V = V_A\left(\frac{N_A}{N_D} - 1\right) \qquad \text{[Eq A2]}$$

where V = volume of distilled water to be added

V_A = volume of solution made up (3 ℓ)

N_A = actual normality (N_{Ag} from Eq A1)

N_D = desired normality (0.5).

*It is recommended that the silver nitrate, sodium chloride, and potassium thiocyanate solutions initially be made slightly stronger by withholding about 10 percent of the water. After titrating, the exact amount of solvent required to produce the 1:1:10 ratio can be computed using Eq A2, and added to the solution.

If the resulting normality of the silver nitrate solution is less than 0.50, additional silver nitrate must be added according to the following equation:

$$W = V_A(N_D - N_A) \qquad \text{[Eq A3]}$$

where W = g-eq wt = (gram-formula wt)/oxidation no. for silver nitrate
g-eq wt of silver nitrate = 169.89 g.

The required solution solvent is added and the process repeated until the desired normality is reached.

3. Sodium Chloride Solution – 0.5*N* (approx)

Weigh out 292.2 g of sodium chloride and dissolve in tap water. Make up 10 ℓ. Standardize against the silver nitrate solution using the Mohr titration technique.

Compute normality (Eq A1) and adjust (Eqs A2 and A3) to produce a 1:1 ratio between the normalities of the sodium chloride and silver nitrate solutions. (One g-eq wt of sodium chloride = 58.448 g)

4. Potassium Thiocyanate – 0.05*N* (approx)

Weigh out 24.3 g of potassium thiocyanate and dissolve in distilled water. Make up 5 ℓ. Standardize against the silver nitrate solution using the Volhard titration technique explained later in this appendix. Compute normality (Eq A1) and adjust (Eqs A2 and A3) to produce a 1.10 ratio between the normalities of the silver nitrate and the potassium thiocyanate. (One g-eq wt of potassium thiocyanate = 97.18 g.)

5. Ferric Alum Indicator

Dissolve 50 g of ferric ammonium sulphate in 100 mℓ of water and add 5 drops of 50 percent nitric acid solution.

6. 50 Percent Nitric Acid Solution

Mix equal volumes of water and nitric acid (concentrated nitric acid 1.41 g/mℓ). Always add acid to water.

7. Nitrobenzene

Use full-strength American Chemical Society (ACS) grade.

8. Mohr Titration Technique (Neutral Solutions Only)

The Mohr method[4] of end point titration can be

[4]Arthur I. Vogel, *A Text-book of Quantitative Inorganic Analysis*, third edition (London: Longmans, 1968), pp 258-267.

used in neutral solutions to determine either the chloride or silver strength if the other is known. The indicator solution is prepared by dissolving 5 g of analytical reagent (AR) potassium chromate in 100 mℓ of water. For a final volume of approximately 50 mℓ of the solution in titration, 10 to 12 drops of indicator solution are required. An acceptable procedure is to pipette 20 mℓ of chloride solution into a 250-mℓ Erlenmeyer flask, adding 10 to 12 drops of potassium chromate indicator solution. Add the silver nitrate solution from a 25-mℓ burette, swirling the liquid constantly, until the reddish brown color formed by the addition of each drop begins to disappear more slowly. This indicates that most of the chlorides have been precipitated. Continue adding drops until a faint but distinct change in color occurs. Persistence of this faint reddish-brown color after brisk shaking indicates that the end point has been reached. The titration should be repeated until two consecutive tests agree within 0.1 mℓ.

9. Volhard Titration Technique

The Volhard method[5] of end point titration is used in acid solutions to determine the reaction between potassium thiocyanate and silver nitrate strengths. For the approximate 1:10 solution concentrations, pipette 5 mℓ of the silver nitrate solution into a 250-mℓ conical flask, add 10 mℓ of 50 percent nitric acid and 5 mℓ of ferric alum indicator solution. Add the potassium thiocyanate solution slowly from a 100-mℓ burette, swirling the liquid constantly, until the reddish color formed by each drop begins to disappear more slowly. This indicates that most of the silver has been precipitated. Continue the addition until a faint but distinct change in color occurs. Persistence of this faint reddish color after brisk shaking indicates that the end point has been reached. The titration should be repeated until two consecutive tests agree within 0.5 mℓ.

[5]Vogel.

APPENDIX B:

PROCEDURE FOR DETERMINING WATER CONTENT WHEN THE SILVER NITRATE: *SODIUM CHLORIDE:POTASSIUM* THIOCYANATE CONCENTRATION RATIO IS NOT 1:1:10

For the standard 50-mℓ titration sample:

$$\% \text{ water} = 50\left(\frac{1 - 0.8a - 2b + 0.02\,bz}{0.8a + 2b - 0.02\,bz}\right)$$

where a = (silver nitrate normality)/(sodium chloride normality)

b = (potassium thiocyanate normality)/(sodium chloride normality)

z = the total volume of thiocyanate in millilitres required to reach end point (blank equivalent plus 50 mℓ plus two times the number of 2-mℓ thiocyanate shots).

For the modified 25-mℓ titration sample:

$$\% \text{ water} = 50\left(\frac{1 - 0.6a - 4b + 0.04\,bz}{0.6a + 4b - 0.04\,bz}\right)$$

where a = same as above

b = same as above

c = the total volume of thiocyanate in millilitres required to reach end point (blank equivalent plus 25 mℓ plus the number of 1-mℓ thiocyanate shots).

APPENDIX C:

WATER CONTENT DETERMINATION—STANDARD PROCEDURE

Sample

1. Place 1-kg concrete sample in the wide-mouth jar.

2. Using volumetric flask, add 500 mℓ of 0.5*N* sodium chloride to concrete sample in jar and seal lid.

3. Agitate concrete and sodium chloride solution mixture for 3 minutes in end-over-end mixer.

4. After agitation, remove jar from mixer and allow contents of jar to settle for 3 to 5 minutes.

5. Using a pipette, obtain 50 mℓ of clear sample from jar and place in conical beaker.

6. To conical beaker add:
   a. 50 mℓ of 0.5*N* silver nitrate solution (automatic pipette)
   b. 10 mℓ of 50 percent nitric acid (fixed volume dispenser)
   c. 2 mℓ of nitrobenzene (fixed volume dispenser)
   d. 5 mℓ of ferric alum (fixed volume dispenser)

7. Using automatic pipette, add 50 mℓ of 0.05*N* potassium thiocyanate to conical beaker. Shake well.

8. Complete titration by adding 2 mℓ shots of potassium thiocyanate from the fixed volume dispenser until the end point is reached (first tint of a permanent reddish-brown color).

9. Record the number of 2-mℓ potassium thiocyanate shots required to reach end point. Use relationship shown in Figure 13 to determine water content.

Blank (Required if Concrete Contains Chlorides)

1. Place 1-kg concrete sample in the wide-mouth jar.

2. Using volumetric flask, add 500 mℓ of distilled water to concrete sample in jar. Seal lid.

3. Agitate concrete and distilled water for 3 minutes in end-over-end mixer.

4. After agitation, remove jar from mixer and allow contents of jar to settle for 3 to 5 minutes.

5. Using a pipette, obtain 50 mℓ of clear sample from jar and place in conical beaker.

6. To conical beaker add:
   a. 10 mℓ of 0.5*N* silver nitrate (automatic pipette)
   b. 10 mℓ of 50 percent nitric acid (fixed volume dispenser)
   c. 2 mℓ of nitrobenzene (fixed volume dispenser)
   d. 5 mℓ of ferric alum (fixed volume dispenser)

7. Titrate by adding 0.05*N* potassium thiocyanate using a 100-mℓ burette until the end point is reached (first tint of a permanent reddish-brown color).

8. Record the millilitres of titrated potassium thiocyanate. Subtract this quantity (mℓ) of potassium thiocyanate from 100 to obtain the number of equivalent millilitres of potassium thiocyanate.

9. Add the number of equivalent millilitres of potassium thiocyanate obtained in step 8 to the number of millilitres of potassium thiocyanate for the sample.

APPENDIX D:

MODIFIED WATER CONTENT TEST

Equipment

The equipment required for the modified water content test is identical to that listed in Table 1 for items 8, 9, and 14. The 50-mℓ pipette and 50-mℓ automatic pipette (items 8 and 9) are replaced with 25-mℓ pipettes. The four fixed volume dispensers (item 14) are replaced with one 1-mℓ, one 2-mℓ, and two 5-mℓ dispensers. The alternate equipment required by the modified procedure has approximately the same cost as the standard equipment.

Procedure

The steps required to conduct a CERL/K-V modified water content test are as follows:

1. Obtain a 6- to 8-kg sample of fresh concrete, mix sample to insure homogeneity, and weigh out two 1-kg subsamples to the nearest gram. Place one 1-kg sample in a wide-mouth jar; using a volumetric flask, add 500 mℓ of distilled water. Secure the lid on the jar. This sample is the blank required for estimating chlorides in the concrete itself.

2. Place the second 1-kg sample in another wide-mouth jar, add 500 mℓ of the approximately 0.5 normal sodium chloride solution, and secure the lid.

3. Secure the two jars in the end-over-end mixer, and mix for 3 minutes.

4. Remove the jars from the mixer and allow contents to settle for 3 to 5 minutes.

5. Using a volumetric pipette, withdraw 25 mℓ of clear sample solution and place it in a 500-mℓ conical beaker. Using the automatic pipette, add 25 mℓ of approximately 0.5*N* silver nitrate solution to the clear sample solution. Using the fixed volume dispensers, add 10 mℓ of 50 percent nitric acid solution (two shots from a 5-mℓ dispenser); 5 mℓ of ferric alum solution (one shot from a 5-mℓ dispenser); and 2 mℓ of nitrobenzene (one shot from a 2-mℓ dispenser). Shake well (by hand) for a few seconds.

6. Determine chloride strength by initially adding 25 mℓ (automatic pipette) of 0.05*N* potassium thiocyanate solution to the solution in the conical beaker. Complete titration using a 1-mℓ fixed volume dispenser containing 0.05*N* potassium thiocyanate solution. Swirl the contents of the beaker while the 1-mℓ shots are added using the fixed volume dispenser. When the first permanent reddish-brown color appears, the end point has been reached. Stop titration and note the number of thiocyanate shots added.*

7. Using a volumetric pipette, transfer 25 mℓ of the blank solution to a conical flask. Add 10 mℓ of silver nitrate solution (automatic pipette), 10 mℓ of 50 percent nitric acid solution (fixed volume dispenser), 2 mℓ of nitrobenzene (fixed volume dispenser), and 5 mℓ of ferric alum solution (fixed volume dispenser). Shake well. Titrate (100-mℓ burette) using the 0.05*N* potassium thiocyanate solution, until the first permanent reddish-brown color appears.

The blank is calculated as follows:

$$y' = 100 - x \qquad \text{[Eq D1]}$$

where y' = millilitres of the thiocyanate equivalent to chloride in the blank

x = quantity of thiocyanate solution in step 7 required to reach end point.

The volume of thiocyanate equivalent (y') obtained from the blank solution is added to the volume of thiocyanate used in the sample solution. The percent water is calculated as follows:

$$\% \text{ water} = \frac{50z}{500 - z} \qquad \text{[Eq D2]}$$

z = two times the total volume of thiocyanate; that is, $2(y' + 25 +$ number of 1-mℓ thiocyanate shots required in step 6).

*The final titration using the 1-mℓ (step 6) dispenser can be accomplished using the 100-mℓ burette, which will give a more exact end point. The choice of the dispenser or burette is the option of the operator.

APPENDIX E:

CEMENT CONTENT DETERMINATION

1. Fill washing machine to mark (10 gal [37.6 ℓ]) with tap water, and place nested sieves on washer. Charge the automatic pipettes with their appropriate reagents—300 mℓ of tap water and 100 mℓ of 5 percent nitric acid.

2. Place 1-kg concrete sample on sieves.

3. Start washer agitation and recirculation pump, and wash cement from aggregate for 1.5 minutes; remove upper sieve (no. 4) and wash cement from aggregate on lower sieve (no. 50) for 1.5 minutes. (If the no. 100 sieve is used, the no. 50 sieve should be removed and the aggregates retained on the no. 100 sieve should be washed for about 1½ minutes).

4. Fill 125-mℓ automatic pipette with cement solution using small hose and clamping large hose.

5. Drain 125-mℓ cement sample and dilute with 100 mℓ of 5 percent nitric acid and 300 mℓ of tap water.

6. Mix above ingredients in stirrer for 3 minutes.

7. Pipette off 25 mℓ of the cement sample and place in 500-mℓ conical beaker.

8. Add 10 mℓ of buffer solution to the 25-mℓ sample, then add four drops of eriochrome black "T" indicator.

9. Fill 100-mℓ burette with .01*M* EDTA solution.

10. Titrate EDTA into cement sample (wine color) until end point is reached. The solution will be a pronounced blue color at the end point.

11. Note millilitres of EDTA used to reach end point; consult calibration graph for the cement being used to determine percentage of cement in mix.

REFERENCES

Howdyshell, P. A., "Correlating Kelly-Vail Test Results to the Strength Potential of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (U. S. Army Construction Engineering Research Laboratory [CERL], May 1975).

Howdyshell, P. A., *Laboratory Evaluation of a Chemical Technique to Determine Water and Cement Content of Fresh Concrete*, Technical Report M-97/AD784005 (CERL, July 1974).

Howdyshell, P. A., *Operations Guide – Water and Cement Content of Fresh Concrete*, Technical Report M-177/ADA022697 (CERL, February 1976).

Kelly, R. T. and J. W. Vail, "Rapid Analysis of Fresh Concrete," *Concrete* (April 1968), pp 140-145.

Vogel, Arthur I., *A Text-Book of Quantitative Inorganic Analysis*, third edition (London: Longmans, 1968), pp 258-267.

MSC

CERL DISTRIBUTION

Picatinny Arsenal
ATTN: SMUPA-VP3

US Army, Europe
ATTN: AEAEN

Director of Facilities Engineering
APO New York, NY 09827
APO Seattle, WA 98749

DARCOM STIT-EUR
APO New York 09710

USA Liaison Detachment
ATTN: Library
New York, NY 10007

US Military Academy
ATTN: Dept of Mechanics
ATTN: Library

Chief of Engineers
ATTN: DAEN-ASI-L (2)
ATTN: DAEN-FEE-A
ATTN: DAEN-FEB
ATTN: DAEN-FESA
ATTN: DAEN-FEZ-A
ATTN: DAEN-MCZ-S (2)
ATTN: DAEN-RDL
ATTN: DAEN-ZCP
ATTN: DAEN-PMS (12)
for forwarding to
National Defense Headquarters
Director General of Construction
Ottawa, Ontario K1AOK2
Canada

Canadian Forces Liaison Officer (4)
U.S. Army Mobility Equipment
Research and Development Command
Ft Belvoir, VA 22060

Div of Bldg Research
National Research Council
Montreal Road
Ottawa, Ontario, K1AOR6

Airports and Const. Services Dir.
Technical Information Reference
Centre
KAOL, Transport Canada Building
Place de Ville, Ottawa, Ontario
Canada, K1A ON8

British Liaison Officer (5)
U.S. Army Mobility Equipment
Research and Development Center
Ft Belvoir, VA 22060

Ft Belvoir, VA 22060
ATTN: ATSE-TD-TL (2)
ATTN: Learning Resources Center
ATTN: Kingman Bldg, Library

US Army Foreign Science &
Tech Center
ATTN: Charlottesville, VA 22901
ATTN: Far East Office

Ft Monroe, VA 23651
ATTN: ATEN
ATTN: ATEN-FE-BG (2)

Ft McPherson, GA 30330
ATTN: AFEN-FEB

Ft Lee, VA 23801
ATTN: DRXMC-D (2)

USA-CRREL

USA-WES
ATTN: Concrete Lab
ATTN: Soils & Pavements Lab
ATTN: Library

6th US Army
ATTN: AFKC-LG-E

I Corps (ROK/US) Group
ATTN: EACI-EN
APO San Francisco 96358

US Army Engineer District
New York
ATTN: Chief, Design Br
Buffalo
ATTN: Library
Saudi Arabia
ATTN: Library

US Army Engineer District
Pittsburgh
ATTN: Library
ATTN: ORPCD
ATTN: Chief, Engr Div
Philadelphia
ATTN: Library
ATTN: Chief, NAPEN-D
Baltimore
ATTN: Library
ATTN: Chief, Engr Div
Norfolk
ATTN: Library
ATTN: NAOEN-D
Huntington
ATTN: Library
ATTN: Chief, Engr Div
Wilmington
ATTN: Chief, SAWCO-C
Charleston
ATTN: Chief, Engr Div
Savannah
ATTN: Library
ATTN: Chief, SASAS-L
Jacksonville
ATTN: Library
ATTN: Const. Div
Mobile
ATTN: Library
ATTN: Chief, SAMEN-D
ATTN: Chief, SAMEN-F
Nashville
ATTN: Chief, ORNED-F
Memphis
ATTN: Chief, Const. Div
ATTN: Chief, LMMED-D
Vicksburg
ATTN: Chief, Engr Div
Louisville
ATTN: Chief, Engr Div
Detroit
ATTN: Library
ATTN: Chief, NCEED-T
St. Paul
ATTN: Chief, ED-D
ATTN: Chief, ED-F
Chicago
ATTN: Chief, NCCCO-C
ATTN: Chief, NCCED-F
Rock Island
ATTN: Library
ATTN: Chief, Engr Div
ATTN: Chief, NCRED-F
St. Louis
ATTN: Library
ATTN: Chief, ED-D
Kansas City
ATTN: Library (2)
ATTN: Chief, Engr Div
Omaha
ATTN: Chief, Engr Div
New Orleans
ATTN: Library (2)
ATTN: Chief, LMNED-DG
Little Rock
ATTN: Chief, Engr Div
Fort Worth
ATTN: Library
ATTN: SWFED-D
ATTN: SWFED-F
Galveston
ATTN: Chief, SWGAS-L
ATTN: Chief, SWGCO-C
ATTN: Chief, SWGED-DC
Albuquerque
ATTN: Library
ATTN: Chief, Engr Div
Los Angeles
ATTN: Library
ATTN: Chief, SPLED-F
San Francisco
ATTN: Chief, Engr Div
Sacramento
ATTN: Chief, SPKED-D
ATTN: Chief, SPKCO-C
Far East
ATTN: Chief, Engr Div
Japan
ATTN: Library
Portland
ATTN: Library
ATTN: Chief, DB-6
ATTN: Chief, FM-1
ATTN: Chief, FM-2
Seattle
ATTN: Chief, NPSCO
ATTN: Chief, NPSEN-FM
ATTN: Chief, EN-DB-ST

US Army Engineer District
Walla Walla
ATTN: Library
ATTN: Chief, Engr Div
Alaska
ATTN: Library
ATTN: NPADE-R

US Army Engineer Division
Europe
ATTN: Technical Library
New England
ATTN: Library
ATTN: Laboratory
ATTN: Chief, NEDCD
North Atlantic
ATTN: Library
ATTN: Chief, NADEN
South Atlantic
ATTN: Library
ATTN: Laboratory
ATTN: Chief, SADEN-TC
Huntsville
ATTN: Library (2)
ATTN: Chief, HNDED-CS
ATTN: Chief, HNDED-SR
Lower Mississippi
ATTN: Library
ATTN: Chief, LMVED-G
Ohio River
ATTN: Laboratory
ATTN: Chief, Engr Div
ATTN: Library
North Central
ATTN: Library
Missouri River
ATTN: Library (2)
ATTN: Chief, MRDED-G
ATTN: Laboratory
Southwestern
ATTN: Library
ATTN: Laboratory
ATTN: Chief, SWDED-TG
South Pacific
ATTN: Laboratory
Pacific Ocean
ATTN: Chief, Engr Div
ATTN: FM&S Branch
ATTN: Chief, PODED-D
North Pacific
ATTN: Laboratory
ATTN: Chief, Engr Div

Facilities Engineer
FORSCOM
Ft Devens, MA 01433
Ft McPherson, GA 30330
Ft Sam Houston, TX 78234
Ft Carson, CO 80913
Ft Campbell, KY 42223
Ft Hood, TX 76544
Ft Lewis, WA 98433
TRADOC
Ft Dix, NJ 08640
Ft Monroe, VA 23651
Ft Lee, VA 23801
Ft Gordon, GA 30905
Ft McClellan, AL 36201
Ft Knox, KY 40121
Ft Benjamin Harrison, IN 46216
Ft Leonard Wood, MO 65473
Ft Sill, OK 73503
Ft Bliss, TX 79916
HQ, 24th Inf, Ft Stewart, GA 31313
HQ, 1st Inf, Ft Riley, KS 66442
HQ, 5th Inf, Ft Polk, LA 71459
HQ, 7th Inf, Ft Ord, CA 93941
West Point, NY 10996
ATTN: MAEN-E
Ft Benning, GA 31905
ATTN: ATZB-FE-EP
ATTN: ATZB-FE-BG
CAC&FL
ATTN: DFAE (3)
Ft Leavenworth, KS 66027
AMC
Dugway, UT 84022
USACC
Ft Huachuca, AZ 85613

AF/PREEU
Bolling AFB, DC 20332

AF Civil Engr Center/XRL
Tyndall AFB, FL 32401

Little Rock AFB
ATTN: 314/DEEE/Mr. Gillham

MSC

Naval Facilities Engr Command
ATTN: Code 04
Alexandria, VA 22332

Port Hueneme, CA 93043
ATTN: Library (Code L08A)
ATTN: Morrell Library

Defense Documentation Center (12)

Washington, DC
ATTN: Bldg Research Advisory Board
ATTN: Library of Congress (2)
ATTN: Federal Aviation Administration
ATTN: Dept of Transportation Library
ATTN: Transportation Research Board

Engineering Societies Library
New York, NY 10017

Oregon State University
Prof. Harold O. Pritchett
ATTN: CERF/Robert Konz
Apperson Hall Room 108
Corvallis, OR 97331

Federal Highway Administration
ATTN: Mr. Doyt Bolling
HHO-33 R 3119
WASH DC 20590

Illinois Dept of Transportation
Bureau of Materials & Physical
Research
126 E. Ash St
Springfield, IL 62706